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Thermal Response of Sapphire to Propellant Combustion

by Mark Bundy

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Army Research Laboratory

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Mark Bundy

Weapons and Materials Research Directorate, ARL

Abstract

Laser ignition is a relatively new approach to initiating the combustion of gun propellant. In this application, the laser pulse is transmitted into the combustion chamber through a window, typically made of sapphire, located in the breech face. In order to evaluate the long-term effects of propellant combustion on the laser window itself, it is important to know the window temperature during firing. This report presents temperature data on a sapphire sample located in the region of the laser window in a laser-ignited 155-mm M199 cannon, firing various charge configurations. It was found, for example, that the sapphire surface temperature peaked at 1,200–1,400° C during the combustion event. It was also found that the surface of the sapphire sample sustained physical damage (cleaving, pitting, and plugging) with each shot fired.

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1. BACKGROUND

Conventional large-caliber guns ignite the main charge in one of two ways. Direct-fire weapons, such as tank guns, typically use a bayonet-type igniter, which consists of a long, perforated metal tube that extends down the central core of the propellant bed. When the electrical firing signal passes through a resistive heating element in the primer head of the igniter tube, it kindles the juxtaposed igniter charge. Jets of hot gas spew from the igniter openings, thereby setting off the main propellant charge. After firing, the stub base, with its long protruding igniter tube, is discarded.

The second type of conventional ignition train, typically used with indirect-fire weapons, such as artillery, is one that utilizes a small breech-end powder charge (base ignition pad) in place of a metal igniter tube. In this system, the firing signal (pin) activates a small primer charge (cartridge) that is externally loaded into the breech block after it is closed. Hot gases, ejected by the primer, impinge on and thereby set off the base ignition pad. Flame-spreading from the base pad throughout the combustion chamber (in some cases, via a center core ignitor) ignites the main charge. There are several concerns associated with this ignition system (Barrows et al. 1993), such as, for a less than a full charge configuration, the free chamber volume will allow the base pad to move beyond the activation range of the primer jet. Furthermore, loading a primer cartridge for each firing is an operation that complicates the design of autoloaders.

Both conventional methods (previously described) ignite the main charge by convective heat transfer, requiring a series of ignition train events to take place. On the other hand, the ultimate goal of laser ignition technology is to use radiation heat transfer to "light" the main charge directly. If laser ignition is successful, it could simplify the combustion process by eliminating some of the mechanical prerequisites and residual ignition hardware that accompanies conventional methods.

In laser-based ignition, radiation is transmitted from an outside laser source into the combustion chamber by means of a fiber-optic cable connected to a "laser window" located in the spindle face of the breech block, e.g., Figure 1. A more extensive account of the laser ignition process can be found in reports by Barrows et al. (1993, 1994). This report deals only with the laser window component, more specifically, with quantifying the window's thermal response to propellant combustion.

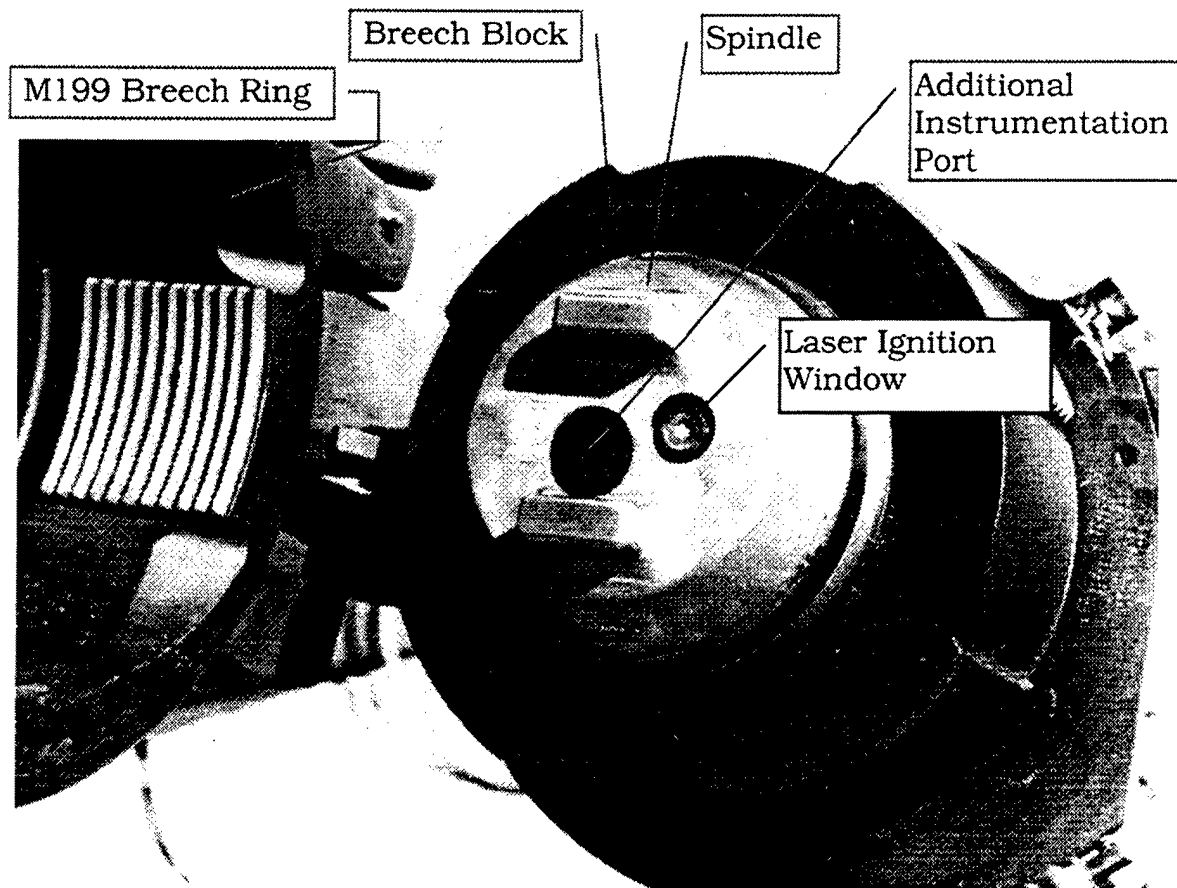


Figure 1. View of the M199 Breech Ring, Breech Block, Spindle, and Experimental Laser Ignition Window.

The laser window must, obviously, be transparent to laser radiation, but it must also be capable of withstanding the high pressures and temperatures generated by the main charge combustion. The material of choice for this application is sapphire (Al_2O_3). Sapphire has a melting point of $2,040^\circ\text{C}$, and is reputed to be chemically stable at high temperatures (Schmid, Khattak, and Felt 1994). It is also high strength, and, at room temperature, it has a

tensile strength along the optical axis of ~425 MPa and a compressive strength of ~1,950 MPa (Harris and Schmid 1995). However, at elevated temperature, its compressive strength may dramatically decrease, incurring a 97-98% loss (to ~50MPa) above 600° C (Harris and Schmid 1995).

Kerwein (1996) noted both cracking and, what appears to be, changes in the surface chemistry on a previously fired laser window. Such findings bring into question whether or not the sapphire surface can reach temperatures high enough to allow mechanical and chemical degradation to take place during the combustion event. Hence, the following experiment was designed to gain information in this area.

2. EXPERIMENT

2.1 PURPOSE. The test objective was to measure the surface temperature of a sapphire sample located where the laser window would be in a 155-mm M199 cannon (e.g., Figure 1) firing various charge configurations.

2.2 INSTRUMENTATION. A current laser window design is shown in Figure 2. It consists of a sapphire crystal, 14 mm in diameter, contained within a 25-mm hexagonal (socket) head. When threaded into the spindle, the sapphire surface of the window is flush with the combustion chamber.

Alongside the laser window in Figure 2 is a thermocouple (TC) probe (K-Type, one of two types) that was used to replicate the window shape and means of attachment to the spindle. A second TC probe type (thin film on sapphire), along with the K-type, is shown threaded in place on the spindle face of an M199 breech block in Figure 3. *

* Both types of thermocouple probes were custom made for this test by Medtherm Corporation, Huntsville, AL.

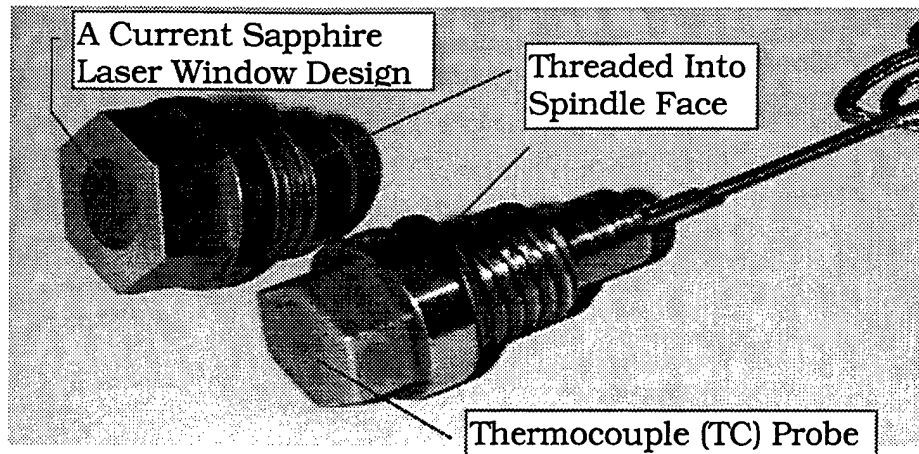


Figure 2. Laser Window and One (of Two) Thermocouple Design(s) Used in Place of the Window to Measure Temperature.

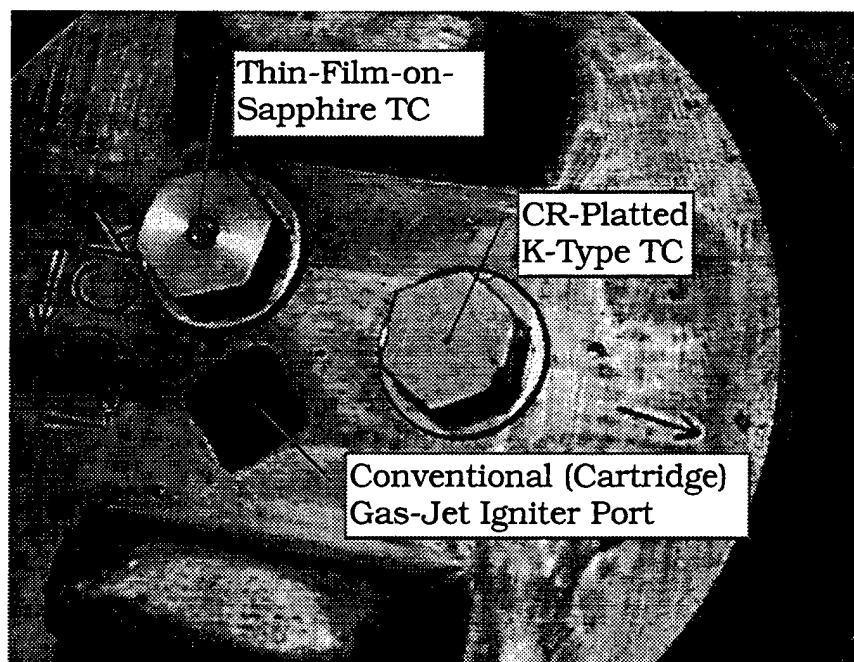


Figure 3. Location of Thermocouples in M199 Spindle Face.

The K-type TC is composed of a central wire of alumel that is separated from an outer coaxial wire of chromel by a thin insulating layer of ceramic. The thermocouple junction was formed by vapor depositing a thin film (1-2 μm) of chromium (Cr) over the sensing end of the coaxial wires, Figure 4.

The thin-film nature of the TC junction gives this type of thermocouple an extremely fast response time, reputed to be on the order of microseconds (Medtherm 1994). Bear in mind, the measured surface temperature is actually that of the probe material itself, which, for the most part, consists of the central alumel wire and surrounding chromel tube. For comparison, the thermal properties (conductivity and diffusivity) of these materials are close to that of the stainless steel housing.

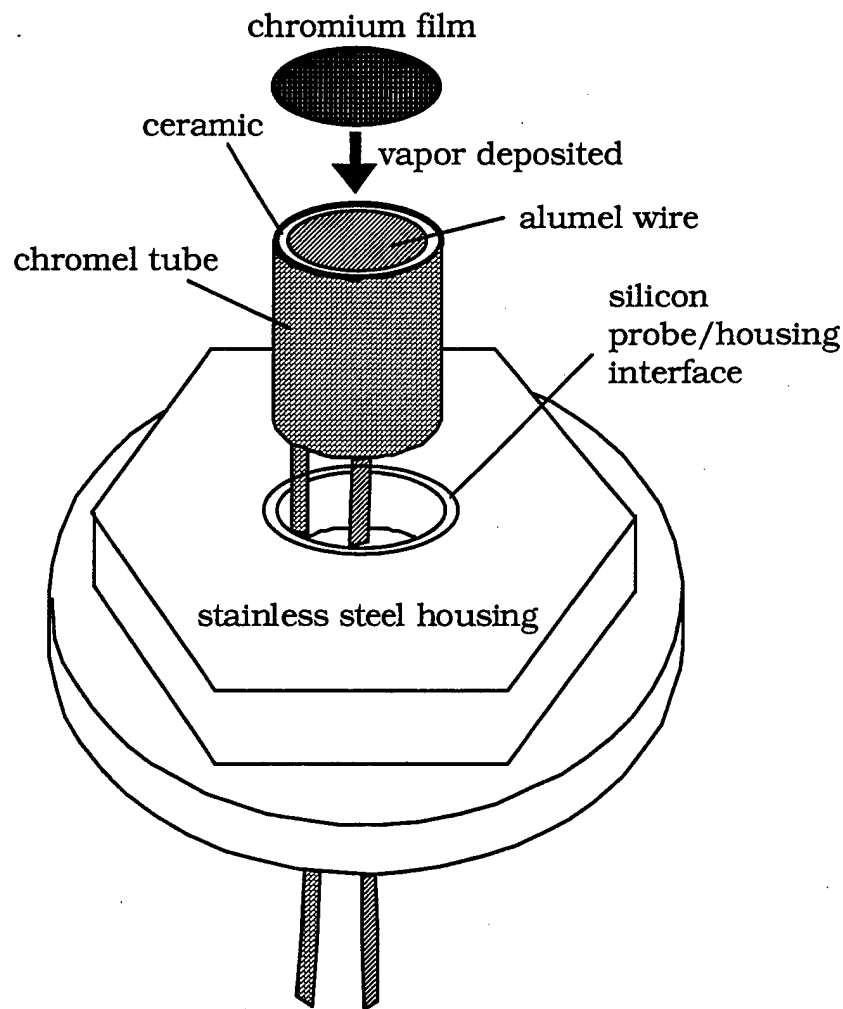


Figure 4. K-Type TC Probe Construction.

The sapphire TC was made from a thin film (0.5–1.0 μm) of rhodium (Rh) and platinum (Pt) vapor deposited on a sapphire crystal, 4 mm in diameter. The thermocouple junction was formed in a central region of thin-film

overlap, as shown in Figure 5. Wires of platinum and rhodium transmitted the temperature-dependent voltage from the bimetallic junction to a signal-processing data recorder. Typical of thin-film TCs, the response time is on the order of microseconds. This sapphire TC provided identical laser window material for duplicating the heat conduction and diffusion effects. Thus, the temperature measurements from this probe should give an accurate assessment of the laser window surface temperature in the gun chamber environment.

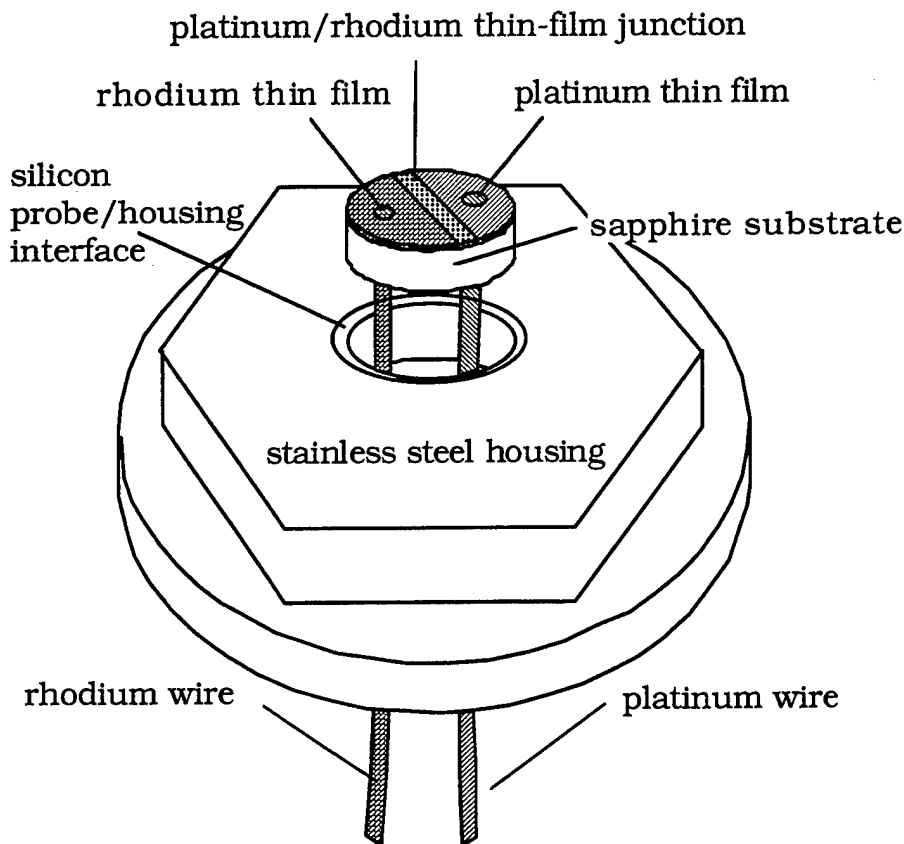


Figure 5. Thin-Film-on-Sapphire Probe Construction.

A broader perspective of the test setup is shown in Figure 6, where the spindle, breech block, and breech ring are included in the view. The thermocouple lead wires extended through and out the back of the spindle, connecting to a data recorder located inside the firing bunker.

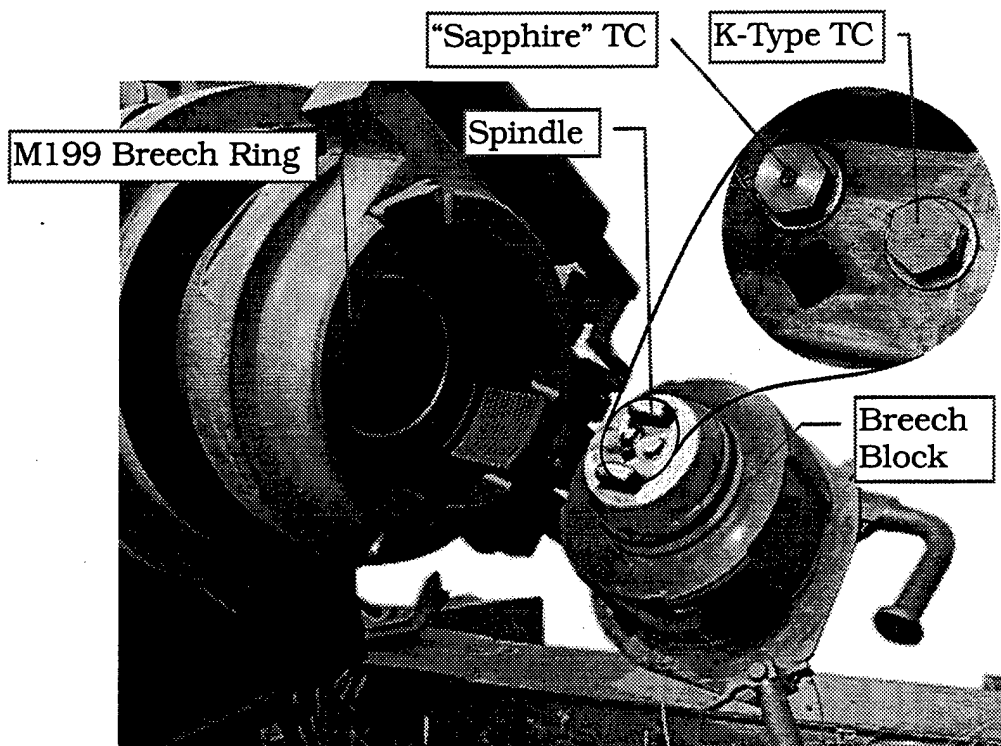


Figure 6. Open-Breech View of TC Probe Locations.

In addition to the TCs, two copper crusher gauges (M11 type) were used to measure the peak internal combustion pressure in the chamber region for each round fired. Also, the muzzle velocity of each round was recorded with a Weibel (Model 680) Doppler radar system. These two sources of information (chamber pressure and muzzle velocity) provided an independent check that the firing was "typical" for a given propellant charge configuration.

2.3 TEST MATRIX. Temperature data were obtained from a ten-round firing program, as described in Table 1. In total, there were five different propellant charge configurations (groups); two rounds of each type were fired. The five groups spanned a range of propellant combustion conditions from low to high charge. The first group, M3A1 zone 5, is made up of five (unequal) increments (zones 1-5) of bagged M1 granular propellant (flame temperature = 2,176° C), totaling 5.5 lb (24.5 N). The second group, M4A2 zone 5, consists of three of a possible five bags (zones 3-7) of M1 granular propellant, totaling 7.1 lb (31.4 N). The third group, M4A2 zone 7, uses all five bags of M1 propellant, totaling 13.3 lb (59.1 N). The fourth group,

M119A2 zone 7, is a single bag of M6 granular propellant (flame temperature = 2,298° C), weighing 20.7 lb (92.1 N). The fifth group, M203A1 zone 8, was a single bag of M31A1E1 stick propellant (flame temperature = 2,301° C) weighing 26.3 lb (117.0 N). The M101 projectile (weighing 96 lb [427 N]) was used for all firings. The M101 is the (obsolete) predecessor of the M107 projectile, having the same weight, the only difference being in the width of the rotating band.

Table 1. Firing Test Sequence

Round Number (Group Number)	Propellant Charge Designation (weight, N)
1 (1)	M3A1 zone 5 (24.5)
2 (2)	M4A2 zone 5 (31.4)
3 (3)	M4A2 zone 7 (59.1)
4 (4)	M119A2 zone 7 (92.1)
5 (5)	M203A1 zone 8 (117.0)
6 (5)	M203A1 zone 8 (24.5)
7 (4)	M119A2 zone 7 (31.4)
8 (3)	M4A2 zone 7 (59.1)
9 (2)	M4A2 zone 5 (92.1)
10 (1)	M3A1 zone 5 (117.0)

3. RESULTS

3.1 CHAMBER PRESSURE AND MUZZLE VELOCITY. The average pressure and muzzle velocity results are tabulated in Table 2. As is typical for this gun system, the peak chamber pressure (averaged over both chamber gauges) was repeatable to within <10% for rounds in the same group. It is also typical for the muzzle velocities to repeat within ~1%, as they did. An instrumentation failure prevented a muzzle velocity measurement for round seven. In general, the chamber pressure and muzzle velocity went up

with increasing propellant charge weight (Table 1) in going from group one to group five. The one exception to this correlation was group two, which had a lower chamber pressure than group one, even though it had more propellant and a higher muzzle velocity than group one. The explanation lies in the fact that the propellant grain geometry was slightly different for the M1 propellant in group two than group one, giving it a different burn-rate history.

Table 2. Maximum Chamber Pressure and Muzzle Velocity Results

Round Number (Group Number)	Charge Designation	Chamber Pressure (MPa)	Muzzle Velocity (m/s)
1 (1)	M3A1 zone 5	112	369
2 (2)	M4A2 zone 5	78	396
3 (3)	M4A2 zone 7	195	569
4 (4)	M119A2 zone 7	223	696
5 (5)	M203A1 zone 8	351	848
6 (5)	M203A1 zone 8	350	846
7 (4)	M119A2 zone 7	223	no data
8 (3)	M4A2 zone 7	181	570
9 (2)	M4A2 zone 5	79	402
10 (1)	M3A1 zone 5	105	371

3.2 BREECH-FACE TEMPERATURES. In general, rounds were fired every 30 min. Probe temperatures were monitored for several seconds prior to and after firing each round. During the combustion event, temperature data were recorded at a 10-kHz rate.

Figure 7 shows a plot of the breech-face surface temperature recorded on the K-type gauge for the first round in the first group. It is speculated that the initial (small) spike, $\Delta T \sim 35^\circ \text{C}$, is coincident with the primer initiation of

the base pad. The peak temperature rise (above ambient) from combustion of the main charge, Δt_{\max} , is $\sim 720^{\circ}\text{C}$, occurring $\sim 70\text{ ms}$ after the primer impulse. For brevity, this will henceforth be referred to as the peak temperature delay time (PTDT). A large part of the temperature rise occurred in a short time span, e.g., the temperature rose 235°C in 1 ms , as shown in Figure 8. It can also be seen from the expanded view of Figure 8 that the temperature remained within 5% of its peak value ($715\text{--}755^{\circ}\text{C}$) for a period of 4 ms . It decayed to 50% of Δt_{\max} in $22\text{--}23\text{ ms}$.

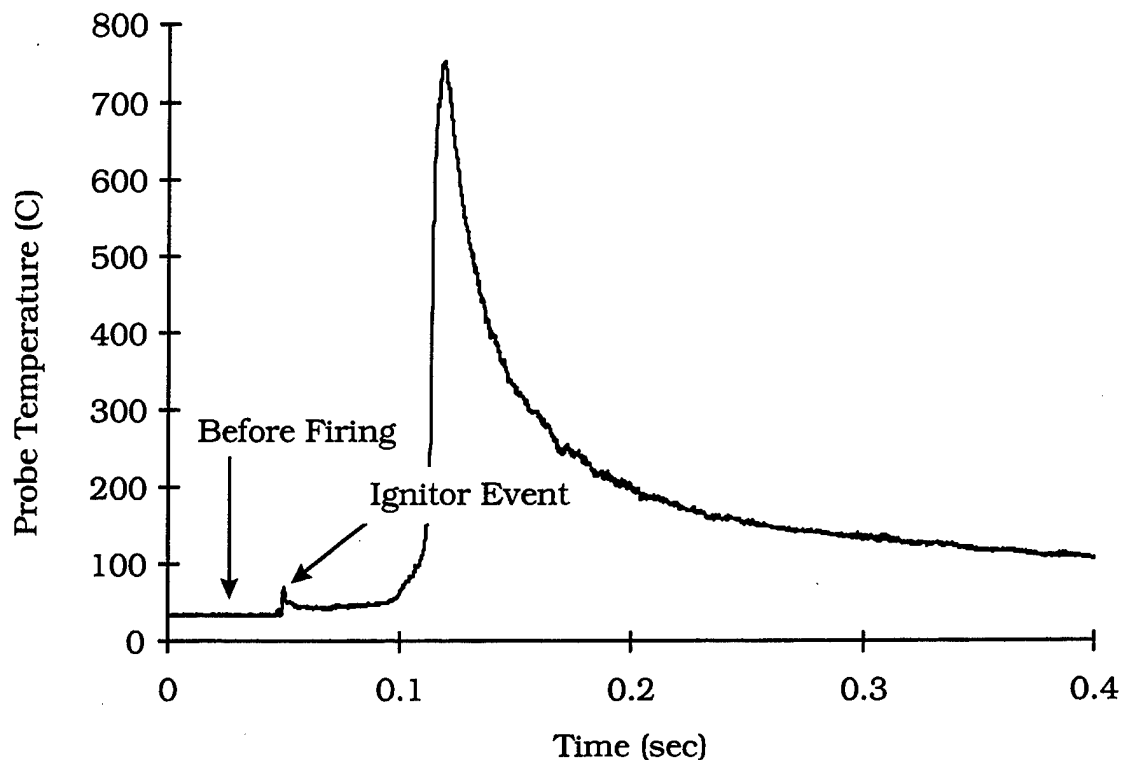


Figure 7. Breech-Face Surface Temperature (K-Type TC) from Firing an M3A1 (Zone 5) Charge.

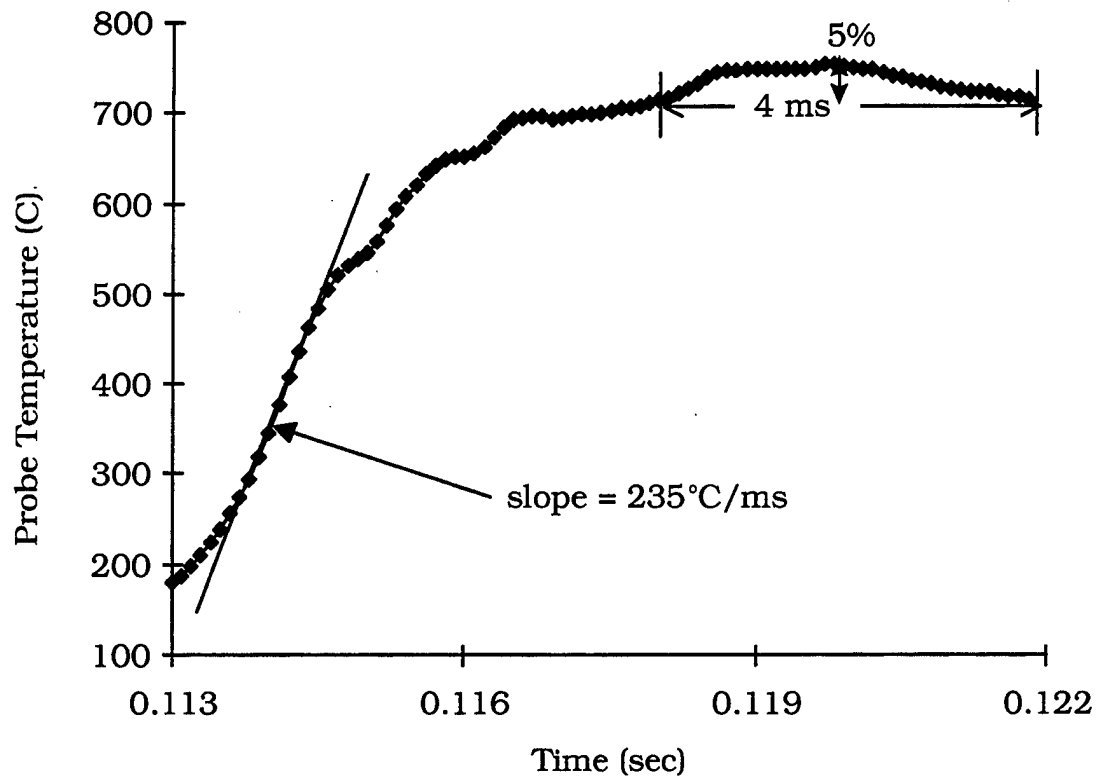


Figure 8. Region of Maximum Temperature Rise, and Peak Temperature, From Figure 7.

Figure 9 displays the temperature time profiles of the first three rounds, as recorded sequentially on the same K-type TC. The relative time scale was adjusted so that the ignitor pulse from all three plots is, more or less, coincident. For the second round, the PTD_T is 90 ms and Δt_{max} is 750° C, while, for the third round, these values are 100 ms and 870° C, respectively. Its compelling to hypothesize that there may be a correlation between increasing peak temperatures and longer PTD_Ts with charge mass, which also increases from rounds one to three. However, it is shown later (Figure 10) that data from other rounds do not support such a correlation.

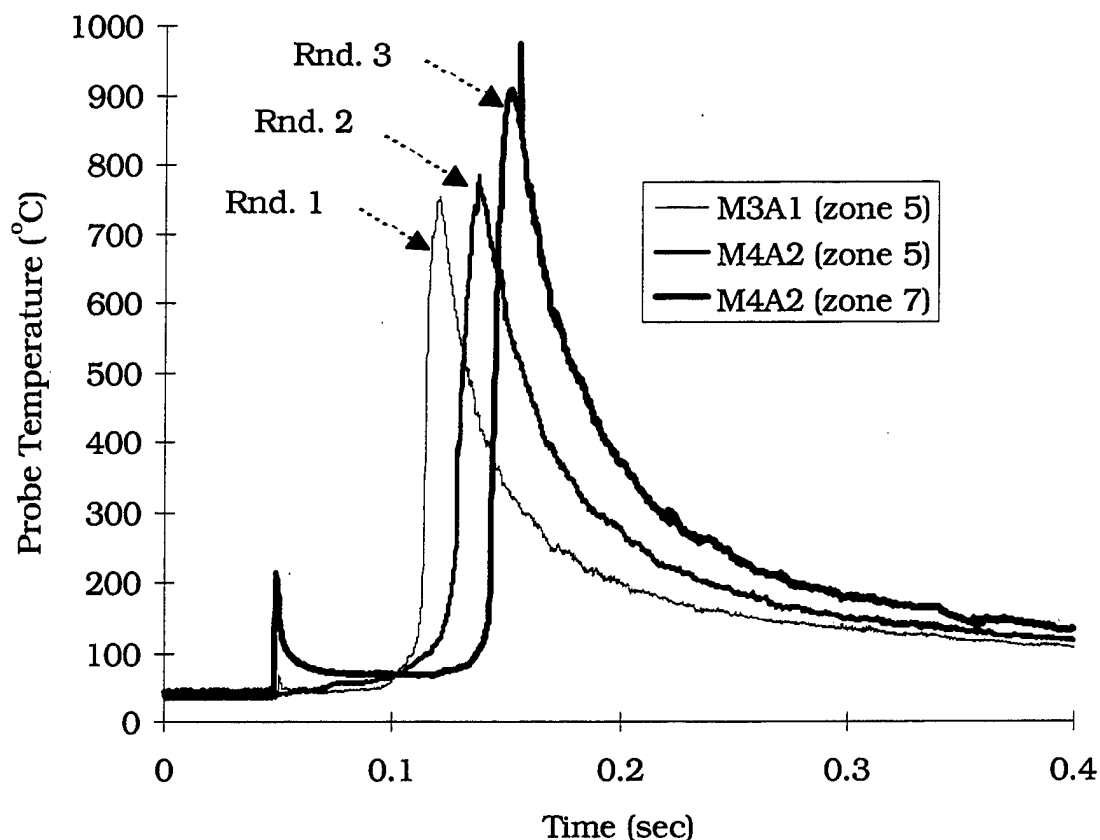


Figure 9. Breech-Face Surface Temperature (K-Type Probe) for First Three Rounds.

For continuity (i.e., before moving on to discussion of the sapphire probe temperature), the surface temperature from the same (K-type) probe for rounds four (M119A2, zone 7) and five (M203A1, zone 8) is shown in Figure 10. As foretold, it can be seen that the progression of higher peak temperatures and longer PTDTs does not continue for rounds four and five. For round four, Δt_{\max} is 830° C and the PTDT is 60 ms, while, for round five, Δt_{\max} is 770° C, with a PTDT of 65 ms; both of which are lower than Δt_{\max} for round three, which was 870° C and 100 ms, respectively. In fact, it is concluded later (Table 3), that at least locally, round-to-round variation in peak temperature and PTDT seems to be a much larger factor than charge mass, charge type (e.g., M1 vs. M6 vs. M31A1E1, or granular vs. stick, etc.), or packaging (e.g., one continuous bag vs. several). This is not to say that the average temperature throughout the chamber, for example, might depend (in

some predictable fashion) on the aforementioned factors. However, before commenting further on round-to-round variation, the K-type probe results, shown thus far, provide some insight into the expected behavior of the sapphire probes.

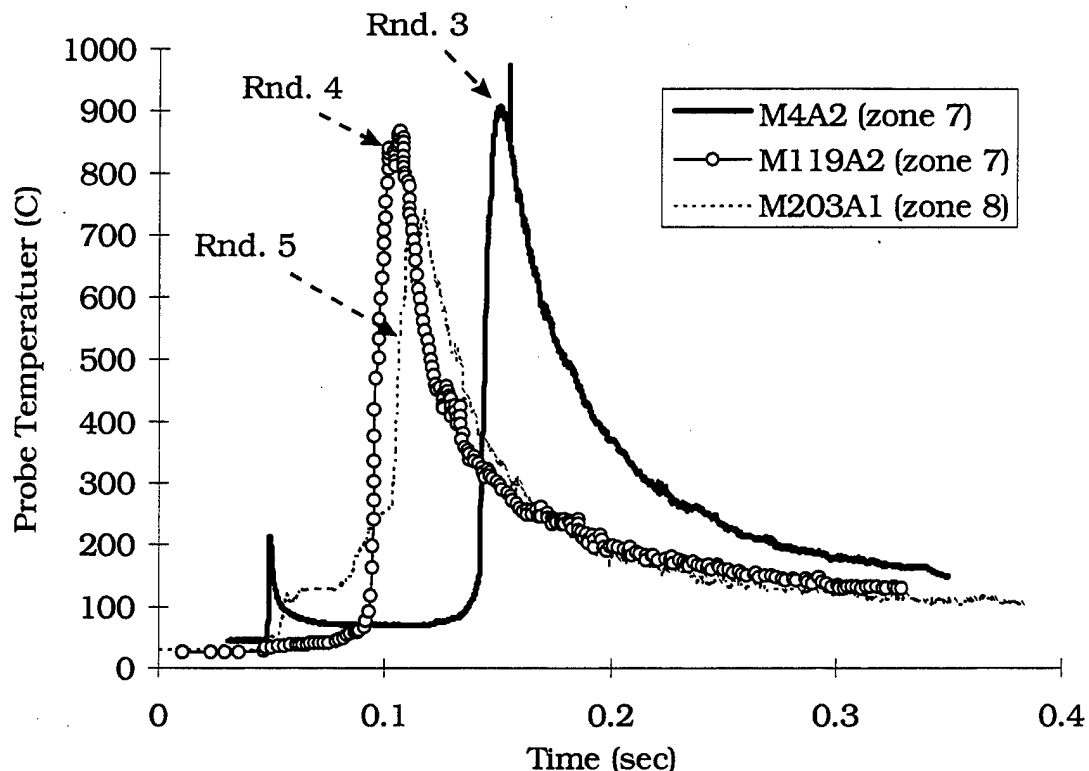


Figure 10. Breech-Face Surface Temperature (K-Type Probe) for Rounds Three, Four, and Five.

Recall that, for each firing, there are two TC probes (Figure 6). One probe is a K-type (Figure 4), and the other is a thin-film-on-sapphire type (Figure 5). It was mentioned in section 1 that there have been measurements reporting that the compressive strength of sapphire along the optical axis drops to ~50 MPa at temperatures above 600° C. Table 2 shows that, in all cases, the peak chamber pressure was ≥ 75 MPa, and, Figures 9 and 10 show that, in all cases, the peak surface temperature exceeded 600° C. Since the thermal conductivity of sapphire is less than that of the K-type probe, the surface temperature of the sapphire probe is expected to be even higher than the K-type probe. If the optical axis of the sapphire TC was, by chance, closely aligned with its axis of symmetry, then the combination of high pressure and

temperature would indicate that the sapphire TC might fail mechanically in this environment. In fact, this may have been the case, since only two of the four sapphire probes procured for this test survived one round (but not the second); the other two probes malfunctioned on their initial firing. Comparatively, the K-type TCs worked well; one K-type gauge operated for nine consecutive rounds.

For the first five rounds, the only round where a sapphire gauge functioned properly was for round three, Figure 11 (the K-type TC temperature is also shown for comparison). As anticipated, the peak sapphire temperature (1,235° C) was higher (320° C) than the K-type probe. Although the absolute temperature values may have been different, both gauges registered the initial temperature spike and the peak temperature at virtually the same time.

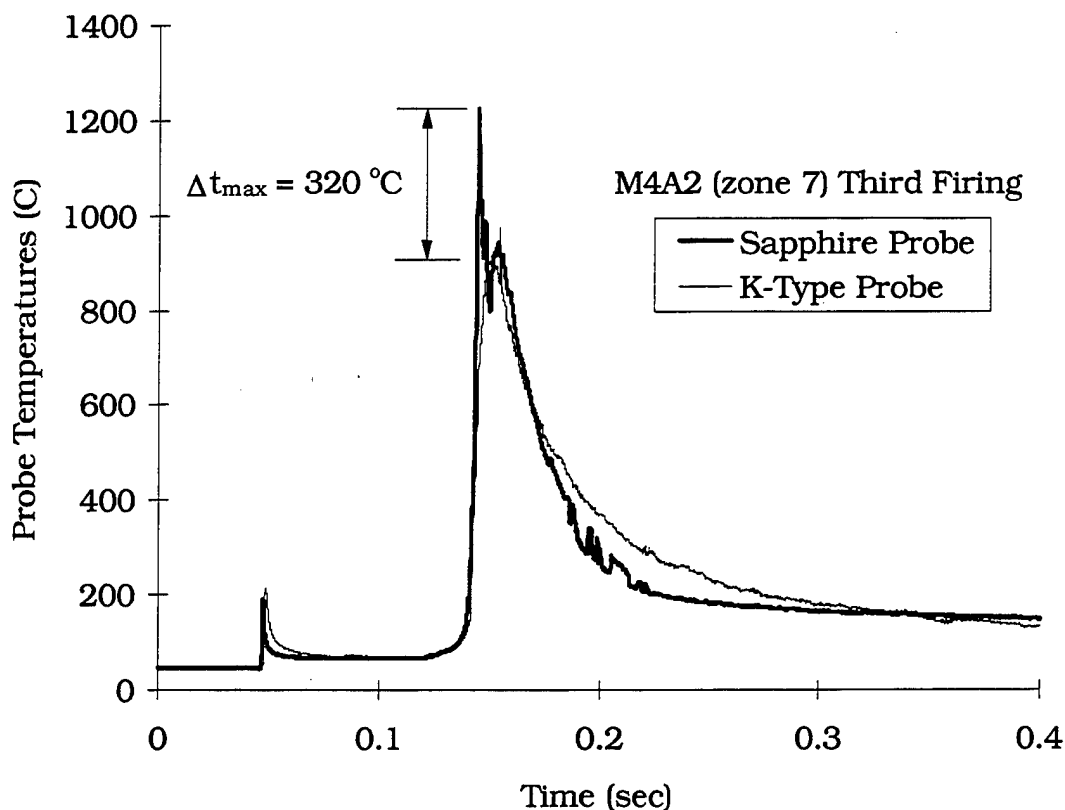


Figure 11. Breech-Face Surface Temperatures of Both Probes for Round Three.

After the third sapphire probe failed, with the firing of only the fourth (of ten) round(s), a K-type TC was threaded into the sapphire port. Thus, for round five, both temperature sensing probes were of the K-type. Figure 12 shows the temperature recorded on the breech face by the side-by-side K-type TCs. It can be seen that the primer pulse and the peak temperatures are very nearly aligned in time and that the peak temperatures differ by only $\sim 55^{\circ}\text{C}$. This 7% difference between adjacent probes of the same type gives some indication that the 30% difference between the K-type and sapphire TCs in round three was due primarily to the difference in their thermal properties.

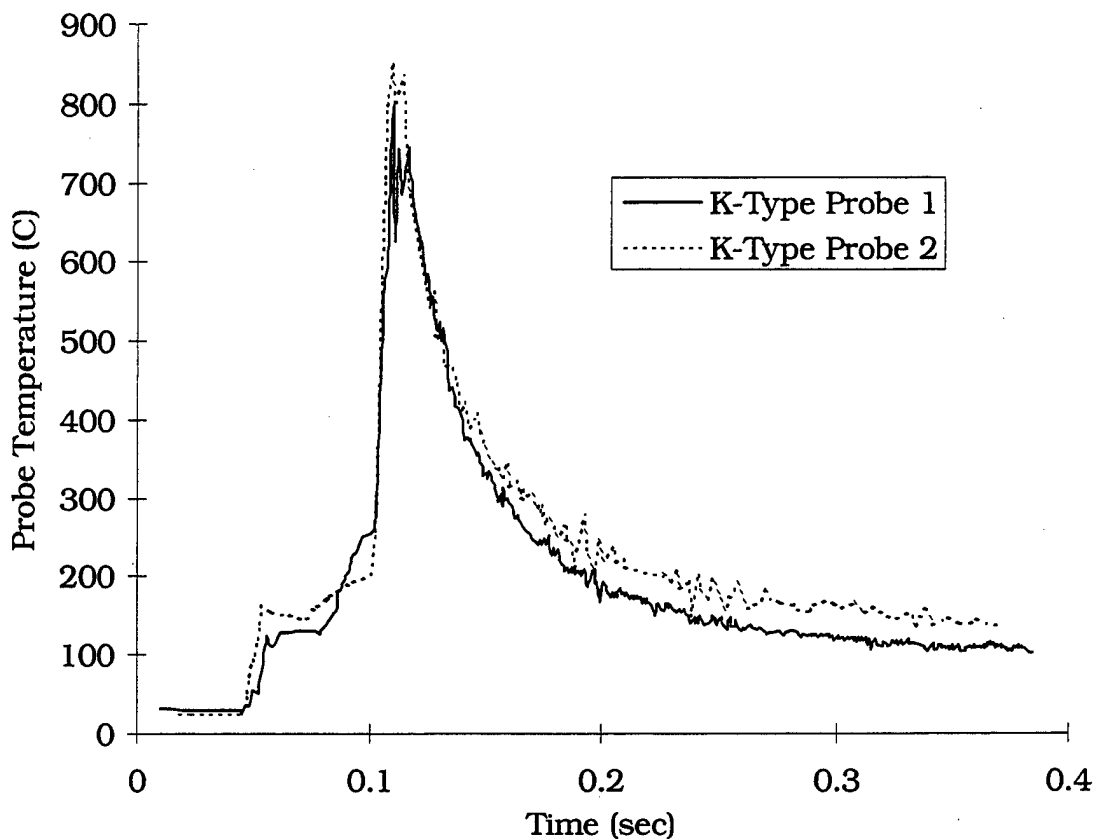


Figure 12. Breech-Face Surface Temperature at Two Adjacent K-Type Probes, for Round Five.

The K-type TC that was used in the sapphire TC port for round five failed on round six. In its place, the last sapphire probe was installed. Fortunately, this probe functioned properly for round seven (though it failed on round eight). Its peak surface temperature was measured to be $1,405^{\circ}\text{C}$, compared

with a reading of 1,020° C from its adjacent K-type probe, Table 3. The same K-type probe that recorded round one, recorded all rounds up through nine, but failed on round ten. Table 3 gives a probe-temperature summary for all ten rounds, along with their corresponding PTDTs.

As alluded to earlier, it is apparent from the scatter in peak temperatures of Table 3, that round-to-round variation is relatively large. For instance, a 55° C probe-to-probe difference was noted in round five, whereas, a 310° C round-to-round difference (measured with the same probe) was noted between rounds two and nine (i.e., between the first and second firing of the M4A2 zone 5 charge). On average, there was about a 185° C temperature difference between the first and second firing of the same charge. At the risk of generalizing from such a small sample size, the round-to-round temperature difference might be as much as three times larger than the probe-to-probe temperature variation.

Table 3. Peak Temperatures and Ignition Delay Times

Round Number (Charge Designation)	K-Type TC (°C)	Sapphire TC (°C)	PTDT (ms)
1 (M3A1 zone 5)	755	TC failure	70
2 (M4A2 zone 5)	785	TC failure	90
3 (M4A2 zone 7)	915	1235	100
4 (M119A2 zone 7)	855	TC failure	60
5 (M203A1 zone 8)	795	850 (K-type TC)	65
6 (M203A1 zone 8)	955	TC failure	65
7 (M119A2 zone 7)	1,020	1,405	60
8 (M4A2 zone 7)	1,030	TC failure	70
9 (M4A2 zone 5)	1,095	TC failure	80
10 (M3A1 zone 5)	TC failure	TC failure	TC Failures

3.3 SAPPHIRE (PROBE) SURFACE DAMAGE. Having implied that the cause of failure in the sapphire TCs was mechanical damage to the crystal from the combination of high temperature and pressure, evidence for this claim is now shown. Figure 13 is a scanning electron micrograph (SEM), magnified 20 \times , of the surface of one of the sapphire TC probes (profiled in Figure 6), as it looked before firing. Although the rhodium and platinum thin-film coatings are not distinguishable in this image, the rhodium and platinum wires running through the sapphire crystal are perceptible.

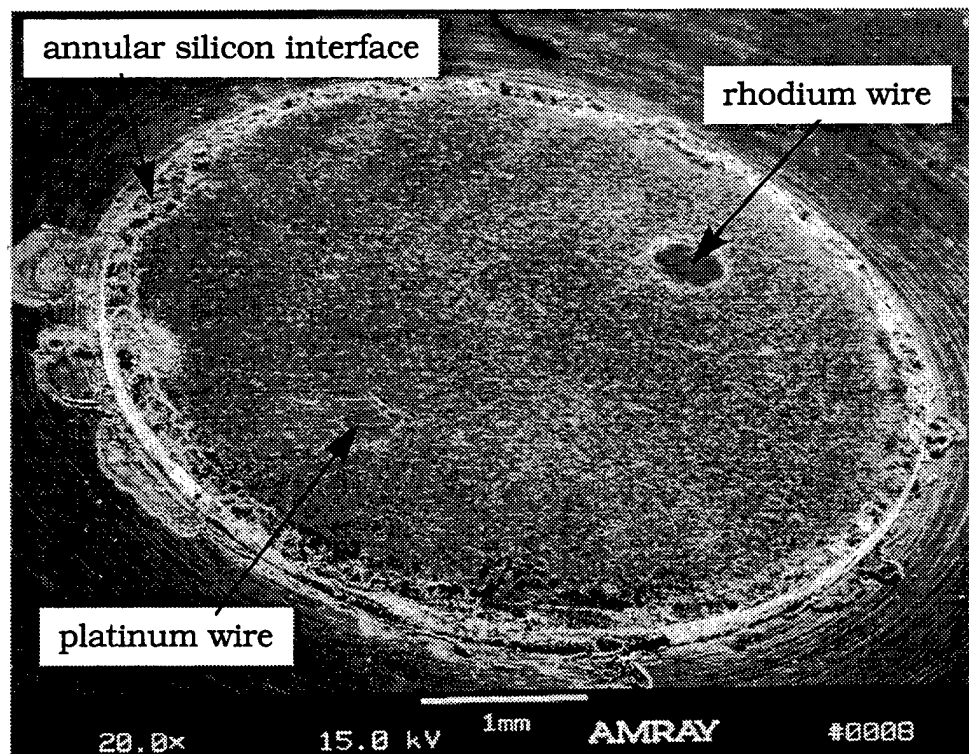


Figure 13. SEM of Sapphire Probe Surface Before Firing.

Figure 14 is an SEM (27 \times) of a sapphire probe after one firing. Clearly, there is physical damage. For instance, the semicircular cleavage line near the outer edge of the probe face is evidence of crystal fracture. Figure 15 provides a close-up SEM view (85 \times) of the fracture site. Also shown in the field of view of Figure 14, but more clearly in Figure 15, is a small crater-like pit, or surface depression in the crystal, roughly 100 μm in diameter.

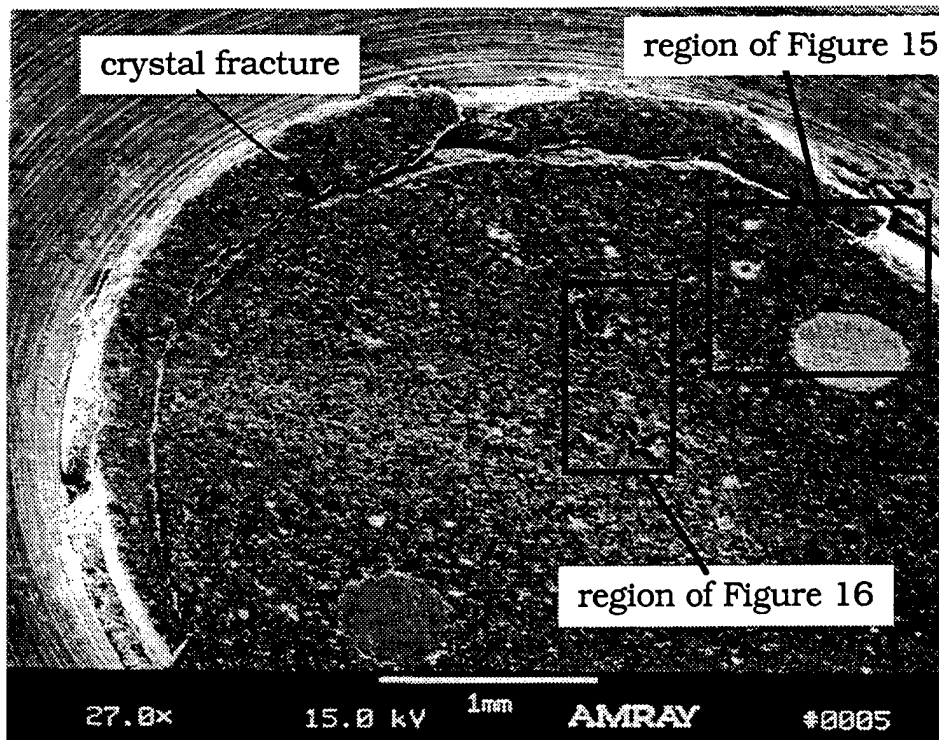


Figure 14. SEM of Sapphire Probe Surface After Firing One Round.

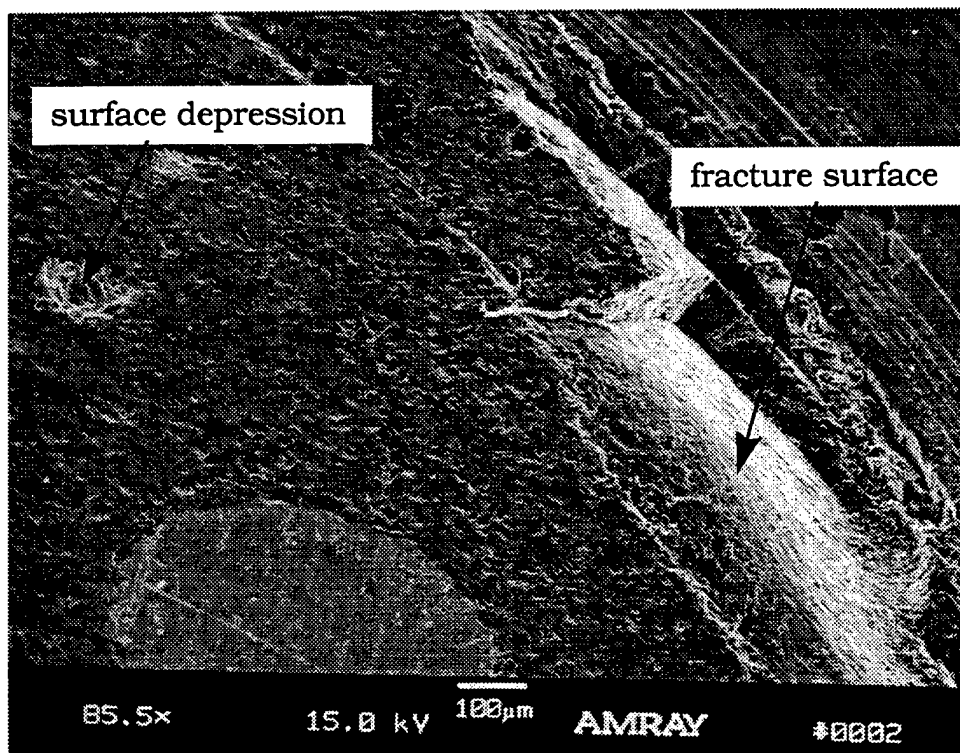


Figure 15 Close-Up SEM of Crystal Fracture, and Crater-like Surface Depression (Magnified From Figure 14).

In addition to fracture and pitting, there are hole-like regions on the crystal surface of Figure 13, magnified in Figure 16, where it appears that a “plug,” or “core” of crystal material is missing. These plugs/cores are 20–100 μm in diameter (depth unknown).

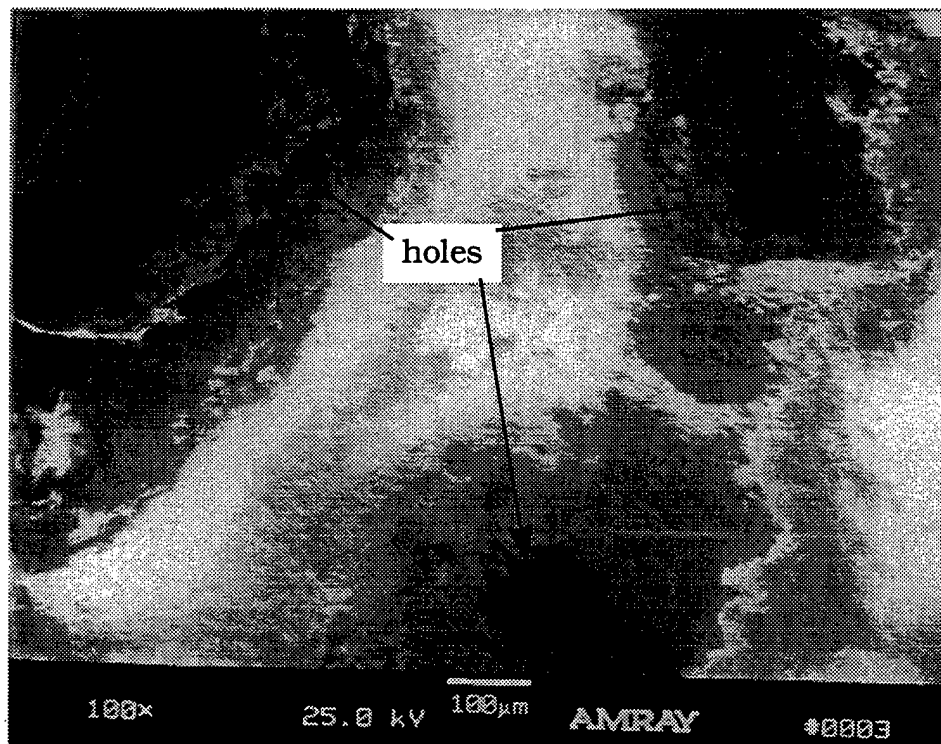


Figure 16. Close-Up SEM of Holes in the Post-Fired Sapphire Surface (Magnified From Figure 14).

It is conceivable that pits and plugs could be caused by the same phenomenon. Reasoning, after peak pressure, the compressed crystal will “spring” back to its original shape. However, bonding in the crystal is weakened by high temperature.* Conceivably, the inertia of the returning surface may exceed the restraining capacity of the bonds in some regions. If so, these regions could break away from the surface, leaving voids that appear as pits and holes. Although speculation on the mechanism of mechanical damage is engaging, it is beyond the scope of this investigation. However, surface chemistry is within this study’s scope and is reported next.

* Harris and Schmid (1995) not only showed that the compressive strength, but also the tensile strength of sapphire decreased with increased temperature on the optical axis.

3.4 ELEMENTAL ANALYSIS OF SAPPHIRE SURFACE. Using an energy dispersive x-ray (EDX) technique, elements (above magnesium, [Mg]) were identified on the surface of the sapphire probe. Before firing, the surface constituents were found to be: aluminum (Al), rhodium (Rh), and platinum (Pt), with trace amounts of sulfur (S), phosphorus (P), silicon (Si), chromium (Cr), and iron (Fe). As expected, the primary element in each half, Figure 17, was either Rh or Pt, with the secondary element being the substrate material—Al. It is not known why there were trace elements of S, P, Si, Cr, and Fe found on the surface.

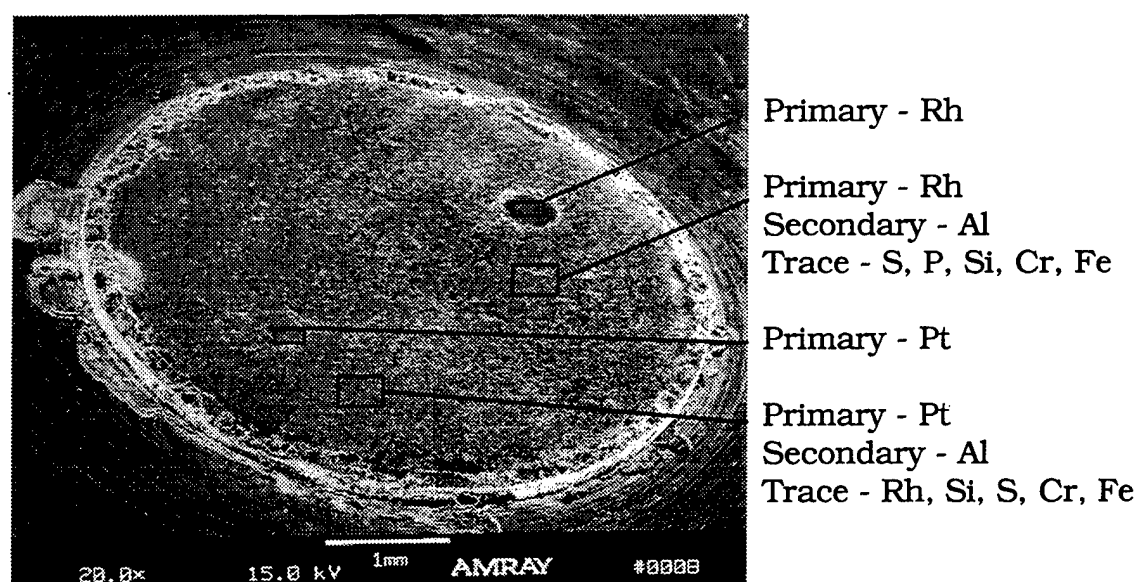


Figure 17. Elemental (EDX) Analysis of Sapphire Probe Before Firing.

After firing, the percentage of surface area that was primarily Rh or Pt decreased dramatically, as shown in Figure 18. Out of five areas sampled sequentially across the Pt-side of the probe, two were found to be primarily Al, rather than Pt. Apparently, a significant fraction of the thin-film coatings of Pt and Rh was removed by the combustion event. A trace amount of potassium (K) was also found on the post-fired surface, this is not unusual, since K (as well as S) is an element of the propellant.

Lastly, in the area of the holes in Figure 16, the primary element was Al, secondary elements were Si, S, K, and P, with traces of Cr and Fe, Figure 19. Noteworthy, Pt is absent, even as a trace element, in these hole regions.

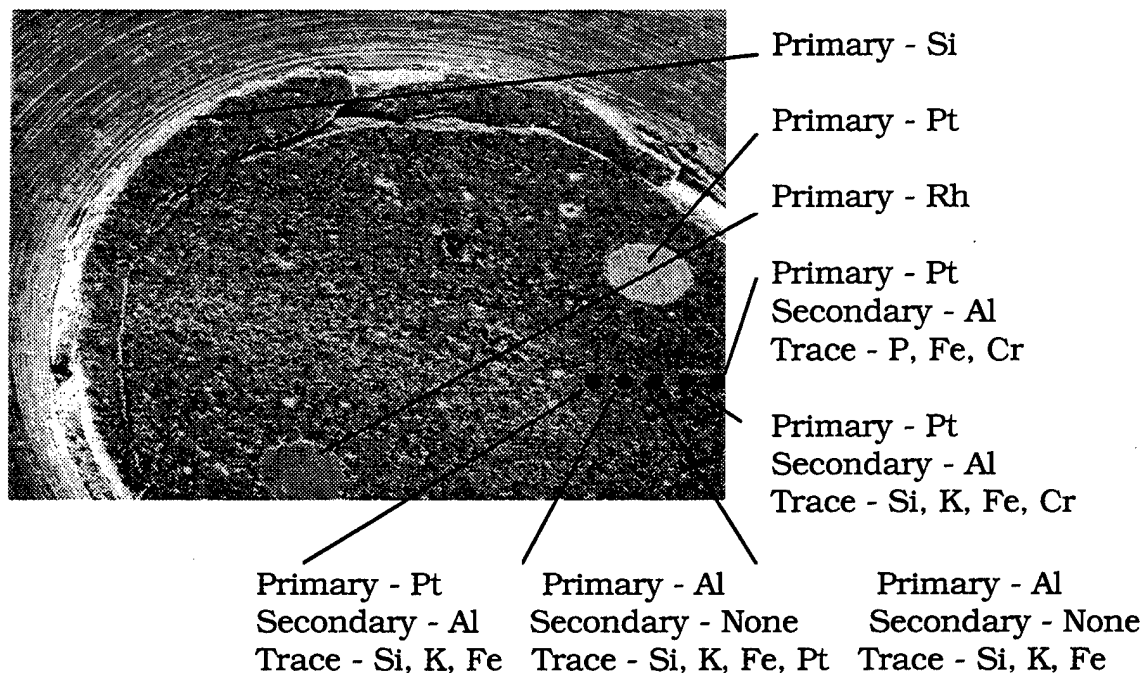


Figure 18. Elemental (EDX) Analysis of Sapphire Probe After Firing.

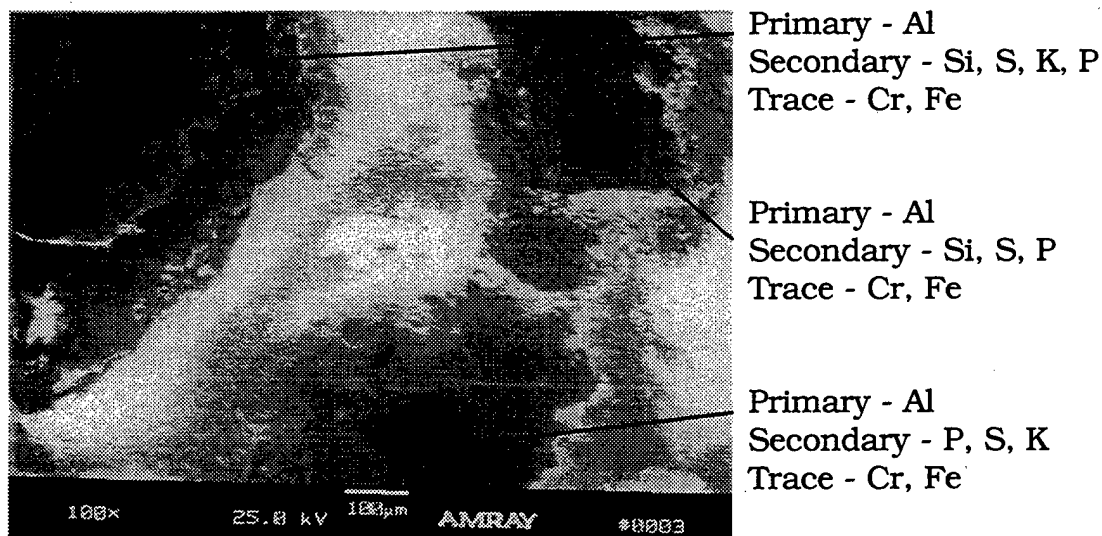


Figure 19. Elemental (EDX) Analysis of Plug Areas on Sapphire Surface.

4. SUMMARY AND COMMENTARY

Sapphire is the window material of choice for laser pulse transmission (through the breech and into the chamber) in laser-ignited guns. However, very little is known about the thermal response of sapphire in this environment. This report expounds upon the test results from an experiment where sapphire was the substrate of a thin-film TC mounted in the same location as a laser window in a 155-mm (M199) cannon. Various charges configurations were fired and the surface temperature measured. The results indicate that a sapphire laser window will incur a peak temperature exceeding 1,000° C in this environment, regardless of charge configuration. (In the two cases where the sapphire probe did not fail, the surface temperature peaked at 1,235° C, for an M4A2 zone 7 charge, and 1,405° C for an M119A2 zone 7 charge.)

Physically, the cylindrically shaped sapphire probe sustained significant damage with each round fired. Semicircular cleaving of the 4-mm-diameter crystal was observed. In addition, there was evidence of surface pitting and plugging, with these features as large as 100 μm in diameter. Although it is not known where the optical axis of the sapphire probe was, in relation to the compression axis, the observed damage would be consistent with a near alignment between the two. Since, in that case, the reported reduction in tensile strength at elevated temperature, coupled with decompression (after the peak chamber pressure is reached) could be the cause of both pitting and plugging on the surface. (Bear in mind, in discussing the possible thermomechanical effects here, it has been tacitly assumed that the peak pressure and temperature occur, more or less, simultaneous. However, in a more detailed failure analysis, it would be important to know the temporal relationship [phase] between pressure and temperature.)

As a commentary, in spite of the rather significant damage incurred by these sapphire samples—after just one firing—sapphire windows have, in general, proven themselves fairly resilient to multiple firings. The explanation for this discrepancy may lie in the details of crystal fabrication for laser windows (vs. thermocouples) and/or in the method of mounting sapphire into a laser window fixture.

Finally, it should be cautioned that the findings of this study, for laser windows in artillery guns, should not be assumed to hold for laser windows in tank gun applications. In artillery, the propellant flame temperature and peak pressure are around 2,300° C and 350 MPa, respectively; whereas, in a tank gun, these values approach 3,200° C and 500 MPa, respectively. This 40% increase in temperature and pressure will, undoubtedly, have a significant effect in the areas reported here.

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